THERMAL AND EVOLVED GAS ANALYSIS OF HYDROMAGNESITE AND NESQUEHONITE: IMPLICATIONS FOR REMOTE THERMAL ANALYSIS ON MARS.

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Introduction: Volatile-bearing minerals (e.g., Fe-oxyhydroxides, phyllosilicates, carbonates, and sulfates) may be important phases on the surface of Mars. In order to characterize these potential phases the Thermal Evolved-Gas Analyzer (TEGA), which was onboard the Mars Polar Lander, was to have performed differential scanning calorimetry (DSC) and evolved-gas analysis of soil samples collected from the surface.

The sample chamber in TEGA operates at about 100 mbar (~76 torr) with a N_2 carrier gas flow of 0.4 sccm. Essentially, no information exists on the effects of reduced pressure on the thermal properties of volatile-bearing minerals. In support of TEGA, we have constructed a laboratory analog for TEGA from commercial instrumentation. We connected together a commercial differential scanning calorimeter, a quadruple mass spectrometer, a vacuum pump, digital pressure gauge, electronic mass flow meter, gas "K" bottles, gas dryers, and high and low pressure regulators using a collection of shut off and needle valves. Our arrangement allows us to vary and control the pressure and carrier gas flow rate inside the calorimeter oven chamber.

Results and Discussion: Hydromagnesite ($Mg_5(CO_3)_4(OH)_2{\bullet}4H_2O$) and nesquehonite ($MgCO_3{\bullet}3H_2O$) are good standard minerals to examine as Mars soil analog components because they evolve both H_2O and CO_2 at temperatures well below the maximum operating temperature of TEGA (<1000 ^oC).

The DSC heat flow and evolved gas curves for hydromagnesite at standard operating conditions (760 torr pressure and 20 sccm N_2) are shown in Figure 1. In order to better understand phase changes during heating, we performed x-ray diffraction (XRD) analyses on samples in which the DSC run was stopped at 360, 470, 525 and 600 ^oC. We found that the first endothermic peak (296 ^oC) corresponds to dehydration, the second (426 ^oC) to dehydroxylation, the sharp exotherm (511 ^oC) to the formation of well-crystalline magnesite and the final endotherm (548 ^oC) corresponds to the dissociation of the magnesite to periclase as CO_2 is evolved.

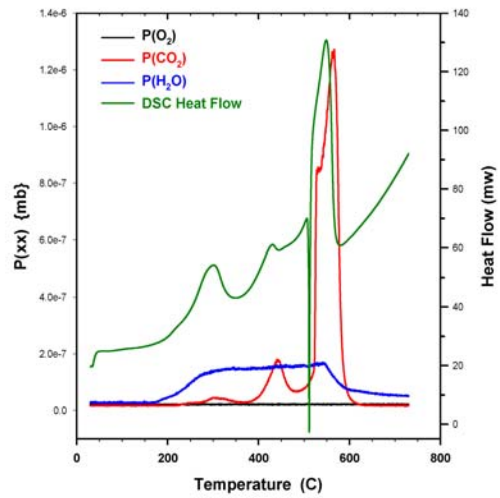

Fig 1: Ambient pressure (760 torr, N_2 carrier gas) heat flow and evolved gas data for hydromagnesite as a function of temperature

The reduced pressure (77 torr @ 12.0 sccm N_2) DSC heat flow and evolved gas curves for hydromagnesite are shown in Figure 2. This time, in order to understand the phase changes during heating at reduced pressure, we performed XRD analysis on samples where the DSC run was stopped at 345 and 520 ^oC. The reduced pressure endotherm (262 ^oC) corresponds to sample dehydration, which resulted in an amorphous $MgCO_3$ phase. The second endotherm (462 ^oC) corresponds to the formation of poorly-crystalline periclase and CO_2 evolution.

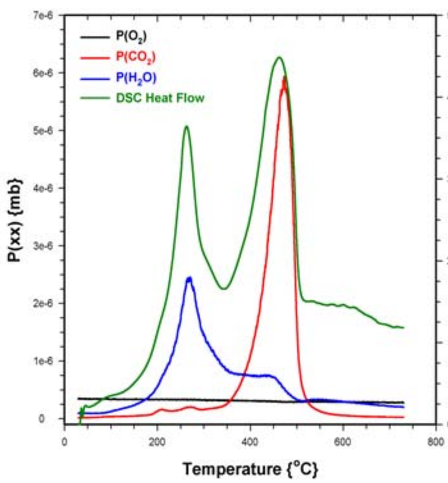

Fig 2: Reduced pressure (77 torr , N_2 carrier gas) heat flow and evolved gas data for hydromagnesite as a function of temperature

Figure 3 shows the results of a 760 torr standard experiment on nesquehonite. The DSC trace and the CO_2 evolved gas curve are quite similar to the reduced pressure results of hydromagnesite. The first endotherm corresponds to sample dehydration to a mostly amorphous $MgCO_3$ phase and the second endotherm (460 ^oC) corresponds to the formation of poorly-crystalline periclase and CO_2 evolution.

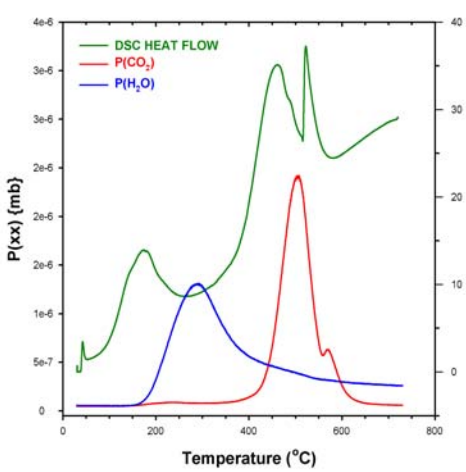

Fig 3: Ambient pressure (760 torr ,N_2 carrier gas) heat flow and evolved gas data for nesquehonite as a function of temperature.

A much different thermal behavior was observed for nesquehonite analyzed in a CO_2 atmosphere (Fig. 4). The CO_2 carrier gas increased the partial pressure of CO_2 in the sample oven that allowed the amorphous $MgCO_3$ phase to form well-crystallized magnesite (the sharp exotherm at 520 ^oC). The final endotherm (600 ^oC) corresponds to the dissociation of the well-crystalline magnesite to periclase as CO_2 is evolved.

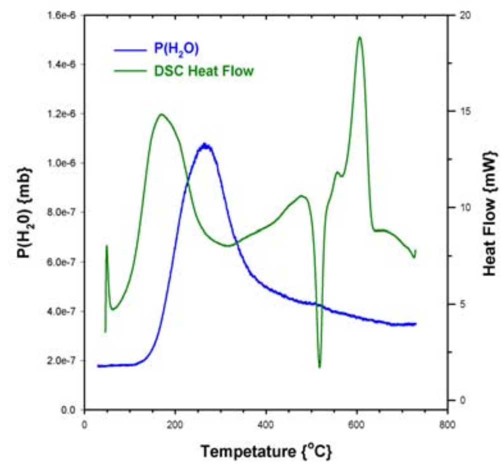

Fig 4: Ambient pressure (760 torr, CO_2 carrier gas) heat flow and evolved gas data as a function of temperature.

SUMMARY: Comparison of the reduced pressure results for hydromagnesite with those taken at ambient conditions show that the effect of heating at reduced pressure can alter the phases forming during heating. In addition the experiments on nesquehonite emphasize the importance of atmosphere composition on the results of a heating experiment.

These experiments emphasize the need of a data base of well-characterized candidate materials analyzed using instrumentation parameters (e.g., reduced pressure on the Mars Polar Lander TEGA) that approach instrument operating conditions on the surface of Mars.